

Lead, atomic absorption spectrometric, direct

Parameters and Codes:

Lead, dissolved, I-1399-85 ($\mu\text{g/L}$ as Pb): 01049

Lead, total recoverable, I-3399-85 ($\mu\text{g/L}$ as Pb): 01051

Lead, suspended recoverable, I-7399-85 ($\mu\text{g/L}$ as Pb): 01050

Lead, recoverable-from-bottom-material, dry wt, I-5399-85 ($\mu\text{g/g}$ as Pb): 01052

1. Application

1.1 This method may be used to analyze water and water-suspended sediment containing at least 100 $\mu\text{g/L}$ of lead. Sample solutions containing more than 4,000 $\mu\text{g/L}$ need either to be diluted or to be read on a less expanded scale. Sample solutions containing less than 100 $\mu\text{g/L}$ need to be analyzed by the atomic absorption spectrometric chelation-extraction method, providing that the interference limits discussed in that method are not exceeded.

1.2 Suspended recoverable lead is calculated by subtracting dissolved lead from total recoverable lead.

1.3 This method may be used to analyze bottom material containing at least 10 $\mu\text{g/g}$ of lead. Prepared sample solutions containing more than 4,000 $\mu\text{g/L}$ need either to be diluted or to be read on a less expanded scale.

1.4 Total recoverable lead in water-suspended sediment needs to undergo preliminary digestion-solubilization by method I-3485, and recoverable lead in bottom material needs to undergo preliminary digestion-solubilization by method I-5485 before being determined.

2. Summary of method

Lead is determined by atomic absorption spectrometry by direct aspiration of the sample solution into an air-acetylene flame.

3. Interferences

Individual concentrations of sodium (9,000 mg/L), potassium (9,000 mg/L), calcium (4,000 mg/L), magnesium (4,000 mg/L), nitrate (900 mg/L), iron (4×10^6 $\mu\text{g/L}$), and cadmium, nickel, copper, zinc, cobalt, and chromium (10,000 $\mu\text{g/L}$) do not interfere. Higher concentrations of each constituent were not investigated.

4. Apparatus

4.1 *Atomic absorption spectrometer* equipped with electronic digital readout and automatic zero and concentration controls.

4.2 Refer to the manufacturer's manual to optimize instrument for the following:

Grating ----- Ultraviolet

Wavelength ----- 283.3 nm

Source (hollow-cathode
or electrodeless-dis-
charge lamp) ----- Lead

Oxidant ----- Air

Fuel ----- Acetylene

Type of flame ----- Slightly oxidizing

4.3 The 100-mm (4-in.), flathead, single-slot burner allows a working range of 100 to 4,000 $\mu\text{g/L}$. Different burners may be used according to manufacturers' instructions.

5. Reagents

5.1 *Lead standard solution I*, 1.00 mL = 200 $\mu\text{g Pb}$: Dissolve 0.2000 g Pb shot in a minimum of dilute HNO_3 . Heat to increase rate of dissolution. Add 10 mL concentrated HNO_3 (sp gr 1.41) and dilute to 1,000 mL with demineralized water.

5.2 *Lead standard solution II*, 1.00 mL = 20 $\mu\text{g Pb}$: Dilute 100.0 mL lead standard solution I and 1 mL concentrated HNO_3 (sp gr 1.41) to 1,000 mL with demineralized water.

5.3 *Lead working standards*: Prepare a series of at least six working standards containing from 100 to 4,000 $\mu\text{g/L}$ of lead by appropriate dilution of lead standard solution II with acidified water. Prepare fresh daily.

5.4 *Water, acidified*: Add 1.5 mL concentrated HNO_3 (sp gr 1.41) to 1 L of demineralized water.

6. Procedure

Aspirate the blank (acidified water) to set the automatic zero control. Use the automatic concentration control to set the concentrations of standards. Use at least six standards. Calibrate the instrument each time a set of samples is analyzed and check calibration at reasonable intervals.

7. Calculations

7.1 Determine the micrograms per liter of dissolved or total recoverable lead in each sample solution from the digital display or printer while aspirating each sample. Dilute those samples containing lead concentrations that exceed the working range of the method and multiply by the proper dilution factors.

7.2 To determine micrograms per liter suspended recoverable lead, subtract dissolved-lead concentration from total-recoverable-lead concentration.

7.3 To determine micrograms per gram of lead in bottom-material samples, first determine the micrograms per liter of lead as in paragraph 7.1; then

$$\text{Pb } (\mu\text{g/g}) = \frac{\mu\text{g/L Pb} \times \frac{\text{mL of original digest}}{1,000}}{\text{Wt of sample (g)}}$$

8. Report

8.1 Report lead, dissolved (01049), total-recoverable (01051), and suspended-recoverable (01050), concentrations to the nearest 100 $\mu\text{g/L}$.

8.2 Report lead, recoverable-from-bottom-material (01052), concentrations as follows: less than 100 $\mu\text{g/g}$, nearest 10 $\mu\text{g/g}$; 100 $\mu\text{g/g}$ and above, two significant figures.

9. Precision

9.1 Precision for dissolved lead for 15 samples within the range of 6.70 to 80.3 $\mu\text{g/L}$ (NOTE 1) may be expressed as follows:

$$S_T = 0.498X - 0.509$$

where

S_T = overall precision, micrograms per liter, and

X = concentration of lead, micrograms per liter. The correlation coefficient is 0.9118.

NOTE 1. Precision data are given for lead concentrations below the reporting level of 100 $\mu\text{g/L}$. Samples were not available that contained greater lead concentrations; however, precision should improve at greater concentrations.

9.2 Precision for dissolved lead for four of the 15 samples expressed in terms of the percent relative standard deviation is as follows:

Number of laboratories	Mean ($\mu\text{g/L}$)	Relative standard deviation (percent)
7	6.70	75
8	25.5	56
13	46.4	51
18	80.3	55

9.3 It is estimated that the percent relative standard deviation for total recoverable and suspended recoverable lead and for recoverable lead in bottom material will be greater than that reported for dissolved lead.

9.4 Precision for total recoverable lead expressed in terms of percent relative standard deviation for two water-suspended sediments is as follows:

Number of Laboratories	Mean ($\mu\text{g/L}$)	Relative standard deviation (percent)
9	44.1	54
7	51.9	38